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FRAGRANCE AGENT

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### Claim

1. A fragrance agent, in which a powder fragrance or a liquid fragrance supported on an organic or inorganic carrier is wrapped in a viscose-processed paper having a heat sealing property.

### Detailed explanation of the invention

#### Industrial application field

This invention concerns a fragrance agent; in more detail, it concerns a fragrance agent which gives a slow releasing performance to the fragrance.

#### Prior art and problems to be solved in this invention

As fragrance devices that are used indoors and in cars, the following are known: those having a structure in which a foam substance immersed in a fragrance solution is stored in a container and sealed in the container having an insertable lid with a supporting shaft attached, it is lifted up and then [the fragrance is] volatilized; spray types in which a fragrance mixed solution is sprayed; and those in which a synthetic resin sheet is immersed in a fragrance, for example.

Fragrances are currently used in many fields, and there are many forms of usage. As one example, many fragrances that are placed in bags are known, and paper and perforated synthetic resin sheets are representative materials of such bags. For example, there is a fragrance agent in which a fragrance is wrapped in a porous sheet with water repellency like polyolefin resins such as polyethylene and polypropylene, for example (Japanese Kokai Patent Application No. Sho 57[1981]-139346); there is a fragrance material in which an inner bag is formed of a thin polyolefin resin film at a thickness of 50-100  $\mu\text{m}$  that is impermeable to liquids and permeable to

gases onto which paper is laminated at the outer face, a fragrance liquid is sealed inside said inner bag, and this inner bag is further sealed into an outer bag, which is formed by laminating a thin synthetic resin film at the inner face or the outer face of a light metallic foil (Japanese Kokoku Utility Model No. Sho 59[1984]-33406), and there is an insecticide and deodorant in which a volatile active substance is wrapped in a wrapping material of which a portion is formed of a porous material, such as Japanese paper and nonwoven fabrics, onto which a ventilation modifier, such as polyethylene, is coated or pasted, and the rest is formed of a transparent nonporous material, such as cellophane (Japanese Kokai Utility Model No. Sho 52[1977]-160680), for example. The use of calcium silicate and/or silica in petal form as a type that is volatilized while adjusting the evaporation speed is also disclosed in Japanese Kokoku Patent No. Sho 59[1984]-27783, however, an excessive amount of a stimulating fragrance is volatilized for a short time or partially at the moment when the lid is released in these methods, allowing the fragrance itself to disappear rapidly.

Moreover, the containers are bulky and expensive, and it is also economically unsuitable to use several kinds of fragrances at the same time for several rooms. From the aspect of a reduction in volume and the aspect of a gradual release accompanied by volatilization over time, they cannot be satisfactorily used.

#### Means to solve the problems

As a result of repeated study to obtain a fragrance agent in which the aforementioned problems are solved and that is economical and easy to handle, the inventors of this invention have discovered that the conventional problems can be solved through the utilization of viscose-processed paper having a heat sealing property, and this invention was completed.

More precisely, this invention offers a fragrance agent in which a powder fragrance or a liquid fragrance supported on an organic or inorganic carrier is wrapped in a viscose-processed paper having a heat sealing property.

As the viscose-processed paper having a heat sealing property used in this invention, there are those that are disclosed in Japanese Kokai Patent Application Nos. Sho 59[1984]-1798 and Sho 59[1984]-1799, in which a recycled cellulose layer is formed on a mixed paper made of hydrophilic fibers and thermoplastic fibers that are hydrophobic and desirably have a heat sealing property, and a manufactured paper, made of a layer of hydrophilic fibers, and a layer of thermoplastic fibers that are hydrophobic and desirably have a heat sealing property, within a range so that the heat sealing property of said thermoplastic fiber does not deteriorate. More precisely, they are obtained by coating at least one side of the aforementioned mixed paper or manufactured paper with a viscose solution by a roll coater, for example, or immersing it,

processing it in a coagulation bath, and then subsequently processing it in a cellulose recycle bath.

As the viscose-processed paper having a heat sealing property used in this invention, an air permeability control type, onto which is laid a cellulose film with small perforated holes weighing 12-70 g/m<sup>2</sup>, desirably 18-60 g/m<sup>2</sup>, and a thickness of 0.02-0.2 mm, is desirable, and one having a proper air permeability can be selected according to the nature of the fragrance and the desired duration period.

When wrapping the fragrance with a viscose-processed paper having a sealing property in this invention, a powder fragrance may be used directly, but a liquid fragrance is used by supporting it on an organic or inorganic carrier. The powder fragrance can also be supported on an organic or inorganic carrier under a proper solvent. As organic or inorganic carriers for supporting the fragrance, polymer particles, such as polyethylene, polypropylene, polyvinyl chloride, polyvinylidene chloride, polyamide, ethylene-vinyl acetate copolymer, and ethylene-methyl methacrylate copolymer, for example, activated carbon, zeolites, silica gel, calcium silicate, and white carbon, for example, can be listed. For supporting the fragrance on a carrier, a carrier and a fragrance are mixed together at a ratio of 99.9-20: 0.1-80, and then sufficiently stirred at a temperature from room temperature to 100°C.

The fragrance can be wrapped in a viscose-processed paper having a heat sealing property by the following method. First, the aforementioned processed paper is mechanically cut into a suitable size in a pair, then three side edges are heat sealed into a bag leaving an opening at one edge. The aforementioned fragrance material in a specific amount is poured continuously into these bags by a filler, then the opening is closed by heat sealing. The fragrance agent obtained in this manner can give the desired gradual releasing property to the fragrance material.

The fragrance agent can be stored for a long period of time by sealing it in an impermeable outer bag, such as cellophane paper and thin vinylidene chloride film, for example, and maintaining it at a low temperature.

#### Application examples

Next, this invention will be explained with preparation examples.

The citrus fragrance KM-1110, floral fragrance KM-1120, and the fruity fragrance KM-1100 that are used in the preparation examples and application examples are prepared in the following formulations.

#### Formulation Example 1

Citrus fragrance KM-1110

Linalool:

4 wt%

Linalyl acetate:	6 wt%
Orange terpene:	30 wt%
Citral:	7 wt%
Lemon oil:	7 wt%
Lime oil terpene:	10 wt%
Lime oil:	4 wt%
Orange oil:	4 wt%
Citronellal:	3 wt%
Lemon terpene:	20 wt%
Cintronellal:	5 wt%
Total:	100 wt%

### Formulation Example 2

#### Floral fragrance KM-1120

Benzyl acetate:	4 wt%
Benzyl alcohol:	8 wt%
Linalool:	8 wt%
$\alpha$ -Hexylcinnamic aldehyde:	2 wt%
Terpineol:	1 wt%
Geraniol:	2 wt%
Nonanediol-1,3-diacetate:	0.5 wt%
$\gamma$ -Decalactone:	0.5 wt%
Tetradecanol:	0.5 wt%
Isoeugenol:	2 wt%
Methyl anthranilate:	0.5 wt%
cis-3-Hexanol:	1 wt%
3-Methyl-3-methoxybutanol:	70 wt%
Total:	100 wt%

### Formulation Example 3

#### Fruity fragrance KM-1100

Methyl 2-methylbutyrate:	2 wt%
Bornyl acetate:	2 wt%
$\gamma$ -Decalactone:	2 wt%
Methyl amyl ketone:	2 wt%
Ethyl benzoate:	3 wt%

Phenylethyl alcohol:	3 wt%
Benzyl acetate:	4 wt%
Ethyl cinnamate:	5 wt%
Ethyl acetate:	5 wt%
Butyric acid:	6 wt%
Ethyl caproate:	6 wt%
Linalool:	8 wt%
$\alpha$ -Terpineol:	8 wt%
Aceto aldehyde diamyl acetol:	10 wt%
Ethyl lactate:	14 wt%
Ethyl 2-methylbutyrate:	20 wt%
Total:	100 wt%

#### Preparation example

To each 10 g of the citrus fragrance KM-1110, floral fragrance KM-1120, and the fruity fragrance KM-1100, which were obtained in the Formulation Examples, 10 g of 3-methyl-3-methoxybutanol (manufactured by Clara K.K., product name: Sorfit) and 10 g of isoparaffin (manufactured by Clara K.K., product name: IP Sorbent 1620) as solvents were added, and respectively placed onto filter paper (8 x 7 cm, thickness 0.35 mm), 50 g of ethylene vinyl acetate copolymer pellets, 10 g of calcium silicate 20-30  $\mu$ m (manufactured by Tokuyama Jyuso K.K., product name: Florite R), and about 10 g of felt (8 x 7 x 0.6 cm), which were prepared beforehand as carriers, while stirring if necessary at room temperature, then placed into a sealable glass container as quickly as possible after the preparation and stored at low temperature.

#### Manufacturing examples

Each kind of carrier supporting 10 g of the fragrance material obtained in the preparation example was sealed into 10 cm long x 8 cm wide bags made of the wrapping papers shown in Table 1. They were placed in sealable glass containers as quickly as possible after the preparation and stored at a low temperature.

Table 1

番号 (1)	包 装 紙 (2)	透 気 度 (秒) (3)	坪 量 (g/cm <sup>2</sup> ) (4)	厚 さ (mm) (5)
1	使用せず (対照) (6)	—	—	—
2	* サフロン (7) 518	482	19.8	—
3	* サフロン 1035	874	37.5	0.064
4	* サフロン 3035	2,880	37.6	0.064
5	* サフロン 5035	5,428	54.6	0.121
6	* サフロン 10060	9,000	62.5	0.099
7	* サフロン	20,000	—	0.063
8	* サフロン	100,000	38.5	—
9	** 皮膜加工品 (8)	なし (9)	45	0.068
10	*** ポリエチレンラミネート紙 (10)	なし	153.0	0.180

- Key:
- 1 Number
  - 2 Wrapping paper
  - 3 Air permeability (seconds)
  - 4 Basis weight (g/cm<sup>2</sup>)
  - 5 Thickness (mm)
  - 6 Not used (comparison)
  - 7 \*Safron
  - 8 \*\*Film processed product
  - 9 None
  - 10 \*\*\*Polyethylene laminated paper

An almost proportional relationship is observed between the air permeability and the number of days duration of the floral fragrance KM-1120 in Table 2. Therefore, it is possible to select the viscose-processed paper having the necessary heat sealing property by anticipating the desired number of days duration and matching this to the air permeability.

\*Viscose-processed paper having a heat sealing property used in this invention  
(manufactured by Fukui Chemical Industry K.K., and by Sansho K.K.)

\*\*Viscose-processed paper to which cellophane paper is laminated

\*\*\*High grade Japanese paper (basis weight 81.7 g/m<sup>2</sup>) onto which a thin polyethylene film at a thickness of 80  $\mu$  is laminated

#### Application Example 1

Of those obtained in the manufacturing example, the citrus fragrance KM-1120 supported on a filter paper was placed into a 100 mL beaker, allowed to stand at room temperature (25°C), and the amount of natural volatilization was measured by weighing on an analytical balance at

set intervals. Table 2 shows the results. The wrapping paper numbers are the same as those in Table 1.

Table 2

① 包装紙番号	② 日 数					
	5	10	21	35	63	84
1	0.71	1.31	2.75	3.55	4.30	4.70
2	0.50	0.95	1.80	2.61	3.62	4.31
3	0.41	0.75	1.41	2.01	2.81	3.52
4	0.35	0.30	1.21	1.71	2.60	3.20
5	0.38	0.57	0.77	1.26	1.94	2.41
6	0.36	0.58	0.80	1.16	1.69	2.10
7	0.22	0.25	0.55	0.95	1.55	1.91
8	0.18	0.22	0.40	0.72	1.23	1.62
9	0.05	0.10	0.16	0.39	0.44	0.67
10	0.16	0.20	0.31	0.50	0.72	0.91

Key: 1 Wrapping paper number  
2 Number of days

#### Application Example 2

Of those obtained in the manufacturing example, the citrus fragrance KM-1110 supported on a filter paper was placed into a 100 mL volume beaker, allowed to stand at room temperature (25°C), and the amount of natural volatilization was measured by weighing on an analytical balance at set intervals. Table 3 shows the results. The wrapping paper numbers are the same as those in Table 1.

Table 3

① 包装紙番号	② 日数					
	5	10	21	35	63	84
1	8.32	9.01	9.82			
2	7.95	8.73	9.75			
3	7.34	8.52	9.61			
4	6.81	8.05	9.32			
5	5.75	7.65	9.01	9.82		
6	5.32	7.21	8.73	9.62		
7	2.48	4.56	7.46	8.94		
8	1.20	2.51	5.15	7.11	9.12	
9	0.27	0.55	1.16	1.56	1.90	2.26
10	2.04	4.02	6.31	8.05		

Key: 1 Wrapping paper number  
2 Number of days

### Application Example 3

Of those obtained in the manufacturing example, the fruity fragrance KM-1110 supported on a filter paper was placed into a 100 mL volume baker, allowed to stand at room temperature (25°C), and the amount of natural volatilization was measured by weighing on an analytical balance at set intervals. Table 4 shows the results. The wrapping paper numbers are the same as those in Table 1.

Table 4

① 包装紙番号	② 日数					
	5	10	21	35	63	84
1	8.31	9.02	9.71			
2	7.92	8.71	9.52			
3	7.60	8.52	9.33	9.90		
4	7.13	8.25	9.12	9.81		
5	6.21	7.41	8.71	9.55	9.62	
6	3.55	4.88	6.63	8.18	9.85	
7	3.03	4.42	5.95	7.25	9.01	
8	2.64	3.87	5.64	6.81	8.01	
9	0.85	1.79	2.60	3.14	3.76	4.31
10	1.47	2.70	4.40	5.91	7.32	8.43

Key: 1 Wrapping paper number  
2 Number of days

### Comparative Example 1

After supporting 10 g of 3-methyl-3-methoxybutanol used as the solvent in the preparation example on a filter paper by the same method as in the preparation example, samples were sealed into bags prepared with each wrapping paper as in the manufacturing example, using the same procedure. Each was placed in a 100 mL beaker, allowed to stand at room temperature (25°C), and the amount of natural volatilization was measured by weighing on an analytical balance at set intervals.

Table 5 shows the results. The wrapping paper numbers are the same as those in Table 1.

From Table 5, it is possible to adjust the days of duration by the viscose-processed paper having a heat sealing property when the amount of polar solvent is excessive.

② Table 5

① 包装紙番号 \ 日数	5	10	21	35	63	84
1	9.35	9.90				
2	8.60	9.61				
3	7.85	9.27	9.92			
4	6.61	8.21	8.65			
5	5.32	6.04	7.93	9.90		
6	2.81	5.41	7.91	9.62		
7	0.75	1.47	2.91	4.69	8.76	
8	0.54	1.03	2.10	3.41	6.13	8.11
9	0.42	0.88	1.80	3.30	5.47	7.30
10	0.11	0.15	0.20	0.24	0.41	0.53

Key: 1      Wrapping paper number  
 2      Number of days

### Comparative Example 2

After supporting 10 g of isoparaffin used as the solvent in the preparation example on a filter paper by the same method as in the preparation example, samples were sealed into bags prepared with each wrapping paper in the same procedure as in the manufacturing example. Each was placed in a 100 mL beaker, allowed to stand at room temperature (25°C), and the amount of natural volatilization was measured by weighing on an analytical balance at set intervals. Table 6 shows the results. The wrapping paper numbers are the same as those in Table 1.

Table 6

②		Table 6						
①	包裝紙番 目 号	数	5	10	21	35	63	84
	1		8.82	9.51				
	2		8.61	9.32				
	3		8.33	9.22				
	4		8.21	9.01	9.91			
	5		7.80	8.73	9.83			
	6		7.54	8.42	9.60			
	7		7.11	8.17	9.51			
	8		4.61	6.52	8.82			
	9		3.82	4.94	7.51	9.21		
	10		0.81	2.61	3.11	4.75	7.30	9.02

Key: 1 Wrapping paper number  
2 Number of days

From Table 6, it can be understood that long periods of desired duration are difficult to achieve without an air permeability over 100,000 sec when the amount of nonpolar solvent is excessive.

#### Application Example 4

Of those obtained in the manufacturing example, 3 carriers on which the fruity fragrance KM-1110 is supported and wrapped in a viscose-processed paper having a heat sealing property (product name: Safron 1035), were each placed in a 100 mL beaker, allowed to stand at room temperature (25°C), and the amount of natural volatilization (g) was measured by weighing on an analytical balance at set intervals. Table 7 shows the results.

Table 7

①	②		5	10	21	35
	担 体	日 数				
③	濾 紙		7.60	8.52	9.33	9.90
④	エチレン-酢酸ビニル 共重合体		7.41	8.33	9.16	9.90
⑤	ケイ酸カルシウム		7.52	8.43	9.24	9.90
⑥	フェルト		7.55	8.46	9.27	9.90

Key: 1 Carrier  
 2 Number of days  
 3 Filter paper  
 4 Ethylene-vinyl acetate copolymer  
 5 Calcium silicate  
 6 Felt

#### Effect of the invention

The fragrance agent in this invention can manifest the desired gradual releasing property by selecting the heat sealing viscose-processed paper according to the nature of the fragrance used and the purpose of use. In addition to the fact that the fragrance agent in this invention is easy to prepare, it is easy to carry. Therefore, it can be effectively utilized in applications such as putting in pockets and placing by pillows, for example, which were not conventional uses, in addition to simply setting it outdoors.